CHARACTERIZATION OF FILM ADHESION BY ACOUSTIC MICROSCOPY

A.K. Mal

Mechanical, Aerospace &
Nuclear Engineering Department
University of California
Los Angeles, CA 90024

R.D. Weglein

Consultant
6317 Drexel Avenue
Los Angeles, CA 90048

INTRODUCTION

Accurate and reliable information on adhesive bond strength between dissimilar solids is an important factor in joint technology. In spite of considerable efforts to obtain a quantitative measure of film adhesion, this parameter remains elusive, particularly so in cases where non-destructive means of determination are desirable if not mandatory.

Non-destructive testing techniques for adhesive bond joints in general have been reviewed in the recent past [1,2]. The lack of adhesion of thin films on substrates [3] as well as the existence of voids [4] have been reported with the reflection acoustic microscope in early diagnostic studies at microwave frequencies.

While this reference work is ample witness to the keen interest in the measurement of adhesion between solid surfaces as well as between films and solids, a reliable measure of bond strength has to date not materialized. The referenced acoustic microscope studies have shown that the complete lack of adhesion, often called a disbond, can easily be detected from acoustic micrograph images. What is not evident to date is the ability of the acoustic microscope to:

a) discern the interface differences existing between two mating surfaces that are somewhere between welded and smooth compressional contact,

b) measure the continuous range of film adhesion strength between the welded contact and the disbond conditions.

A quantitative technique is consequently desired by which the bond strength at an interface can be characterized and measured simply, rapidly and with cost effectiveness. Layer-guided Rayleigh and Lamb-type waves appear to be particularly attractive in this regard, because:

a) their characteristic particle motion exhibit both normal and tangential components with respect to the interface plane where the bond strength is to be ascertained, and

b) the metrology mode of the acoustic microscope [4], that yields the Rayleigh velocity quantitatively, is indeed cost effective as compared to the imaging mode.

Since guided waves are inherently generated in the wide-angle lenses used in the acoustic microscope [4], it would seem worthwhile to study its use for adhesion strength with that objective in mind. We briefly review the relevant features of the acoustic microscope instrumentation before describing the experiments on layered samples and their preliminary interpretation. Next, we present computed wave dispersion curves for the two lowest modes that may propagate in this particular regime and conclude with a comparison of the measured and computed results.

Description of Experimental Specimens

The experimental specimens were prepared with processes that would predict good as well as substandard adhesion between top layer (Ti) of thickness h and the substrate (Be) of thickness 2mm. The three samples were designated:

Class I: predicted "good", based on a proven surface preparation and deposition process to yield historically reliable adhesion, $h \simeq 42$ µm;

Class II: predicted "bad", in which the Ti coating was carried out with low deposition temperature, $h \simeq 39$ µm;

Class III: predicted "bad", in which the Be substrate was wet machined prior to the deposition, $h \simeq 44$ µm.

Description of Acoustic Microscope

Although the acoustic microscope at Lawrence Livermore National Laboratories (LLNL), on which these experiments were carried out, has been previously described [5], a brief review is appropriate here for the sake of continuity. The LLNL acoustic microscope facility is unique in at least five distinct aspects:

(a) An ultra broad (10 to 100 MHz) frequency range is provided through two frequency-contiguous Quartz/$LiNbO_3$ transducer/lens assemblies.

(b) Two signal source options are available through which coherent RF pulse burst or quasi-coherent video impulse excitation may be selected.

(c) Precision imaging (color as well as black and white) and metrological algorithms may be implemented through digital control.

(d) A large digital storage capacity is available which permits near real-time replay of in-process work.

(e) A large library of historical files of images and metrology data is available on-line for review.

These wide-angle acoustic lenses, used almost exclusively in acoustic microscopy, excite Rayleigh waves and other surface skimming modes at the solid/fluid interface. It is for this reason that the unique properties of Rayleigh waves, described above, are intimately related to the metrology mode or Acoustic Material Signature (AMS) mode of the acoustic microscope [6].

The 10:1 frequency range of the system permitted the measurement of the film thickness of layered substrates [7] and the investigation of surface regions modified through machining damage [8]. Rayleigh wave dispersion is of particular interest to the subject of this paper where a wide frequency range is required to obtain an accurate measure of Rayleigh wave dispersion.

Measured Results

The specimen geometry and the bulk properties of its constituents are described in Figure 1 and Table 1. Dispersion measurements were carried out in the acoustic microscope in the frequency range from 23-40 MHz and the results are shown in Table 2.

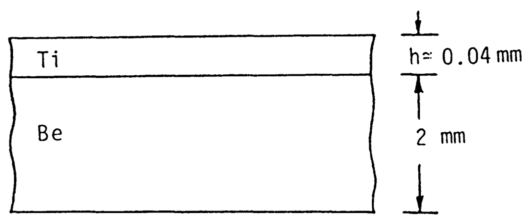

Figure 1. The specimen Geometry

Table 1. Properties of Component Materials

Material	Thickness h (mm)	P-wave velocity α (mm/μsec)	S-Wave velocity β (mm/μsec)	Rayleigh wave velocity v_R (mm/μsec)	Density ρ (gm/cc)
Ti	.039-.044	6.13	3.18	2.96	4.54
Be	2	12.80	8.88	7.86	1.85

Table 2. Measured SAW velocity from AMS data.

F(MHz)	Velocity (mm/μsec)		
	I	II	III
23	6.39	6.71	5.82
27	4.27	6.22	3.39
35	3.54	5.41	5.25
40	2.98	3.39	5.09

THEORETICAL MODELING

In this section we discuss the computation of the velocity of guided waves in the layered specimen with or without perfect adhesion at the interface.

We first consider a theoretical model in which the Titanium layer of thickness h is perfectly bonded to the Beryllium substrate. The thickness of the interfacial bonding layer is assumed to be negligibly small compared to all other length dimensions. In addition, it is assumed that the layer guided waves have wavelengths that are small compared to the thickness of the substrate in the frequency range of interest, so that the substrate may be modeled as a semi-infinite medium. It is well known that the resulting single-layered half space can support dispersive Rayleigh waves whose velocity-frequency relation may be expressed in the form

$$\Delta(v_R, F, h) = 0 \tag{1}$$

where v_R is the phase velocity in mm/μsec, F is the frequency in MHz and h the layer thickness in mm.

The expression for Δ in equation (1) must be obtained through the solution of a boundary value problem of elastodynamics with appropriate boundary, interface and radiation conditions. The real roots, v_R of equation (1) for given frequencies and material parameters can then be obtained through standard techniques. This problem has been of great interest to seismologists for many years and the necessary analysis and highly efficient computer codes have been developed by them to calculate the phase velocity, group velocity and displacement-depth profiles of Rayleigh waves propagating in multilayered half spaces. Detailed accounts of these techniques can be found in the standard seismological literature (see, e.g., Aki and Richards [9], Kundu and Mal [10]) and will not be repeated here.

We have developed a computer program based on similar theories to calculate the phase velocity of guided waves in multilayered solids in the frequency and material parameter ranges of interest in ultrasonics. The solid may be semi-infinite, giving rise to Rayleigh waves, or of finite thickness, giving rise to Lamb waves. The details of these computations can be found in [11, 12 and 13]. We have used these programs to conduct a series of numerical experiments in an effort to interpret the data shown in Table 2.

Our first model consists of a Titanium layer welded to a Beryllium substrate. For a .04 mm thick layer, the calculated Rayleigh wave velocities in the case of a semi-infinite substrate and the Lamb wave velocities in the case of a 2 mm thick substrate in the frequency range of interest are shown in Table 3. It can be seen that the velocity of the first two wave modes are very nearly equal in the two cases. The effect of the bottom surface of the plate is the presence of a third mode, which is not shown and will be ignored in the present analysis. The results shown in Table 3 support the intuitive argument that the substrate can indeed be modeled as a semi-infinite medium.

The calculated Rayleigh wave phase velocities for the three Titanium layer thicknesses involved in the measurements are shown in Fig. 2 together with the measured velocities. It can be seen that the measured velocities in the "good" specimen are in fairly good agreement, except for one data point, with the calculated velocity of the fundamental Rayleigh

Table 3. The effect of the bottom of the substrate.

Frequency (MHz)	Phase Velocity (mm/μsec)			
	Semi-infinite		2 mm Substrate	
23.000	5.01	7.58	5.01	7.58
27.000	4.60	6.67	4.60	6.67
31.000	4.11	6.10	4.11	6.10
35.000	3.70	5.80	3.70	5.81
39.000	3.44	5.62	3.44	5.62

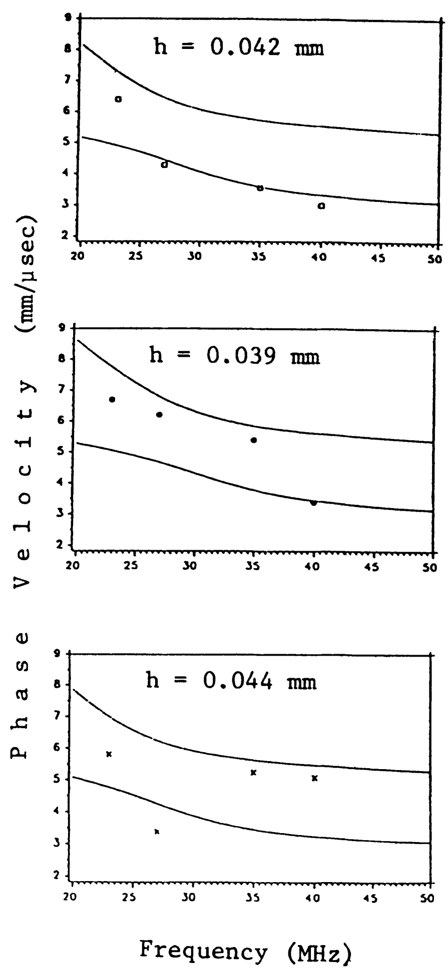

Figure 2. Calculated dispersion curves under the assumption of welded interface for the three specimen thicknesses

mode. However, the data for the "bad" specimens are closer to, but not in very good agreement with the calculated first higher mode velocities.

Examination of Fig. 2 raises a number of interesting questions that deserve comment. To begin with, the significant differences in the measured velocities in the "good" and "bad" specimens seem to suggest that the leaky waves produced in the acoustic microscope experiment have been influenced by materials at different depths in the specimens. For the "good" specimen, the leaky waves are associated with the fundamental mode Rayleigh waves which scan the upper part of the medium with a lower average wave speed. This is the generally accepted result for a layered half space with a welded interface. The measured high velocities in the "bad" specimens imply that the leaky waves are associated with waves which propagate at lower depths and are therefore more influenced by the substrate. This leads to the natural conclusion that the welded interface model must be modified to account for this possibility in the theoretical calculations.

In order to construct a reasonable model of the interface for the "bad" specimens we recall that for imperfect bonding it is possible, indeed probable, that the continuity conditions for the displacement vector across the interface may be violated for these specimens. The sample preparation for Class II and Class III specimens also implies that their interfaces may contain a thin layer of a material which may have different properties from those of the constituents. Accordingly, this interfacial layer will now be incorporated in our theoretical model. Since the mechanical properties of the interfacial layer are not known a priori, we considered a series of models in which the thickness, density, and the elastic moduli of the layer were varied in a wide range and our program was used in each case. The results are summarized in Figs. 3a and 3b.

In Fig. 3a, the calculated Rayleigh wave dispersion curves are shown for varying properties of the interfacial layer. The thickness of this layer is 100th that of the Titanium layer and its shear velocity is reduced from that of Titanium by an order of magnitude. It should be noted that the decrease in the shear modulus of the material is two orders of magnitude since its density is kept constant. The frequency is normalized so that the results are independent of specimen thickness. It can be seen that the phase velocity of both Rayleigh modes are very strongly affected in a limited frequency range, in spite of the fact that the wavelengths of the Rayleigh waves in this range is much larger compared to the thickness of the interfacial layer. It was found that further reduction in the shear velocity of the layer material has no effect on the Rayleigh wave phase velocity in the frequency range of interest ($20 < F < 40$ MHz). This is more demonstrated in Fig. 3b which shows the influence of the reduction in shear velocity in the interfacial layer on the Rayleigh wave phase velocity in the frequency range of interest. Clearly, the limiting case of zero shear in the interfacial layer is reached as its shear wave velocity becomes lower than that of the Titanium layer by a factor of 10. This implies that the lowest curves in Fig. 3a corresponds to the so called "oil-coupled" interface or "kissing contact" in which the normal components of the displacement and stress vectors are continuous, the tangential component of the stress vector (i.e., the shear stress) is zero and the tangential component of the displacement vector is discontinuous.

We now return to the interpretation of the data. To this end all the measured data are shown in Fig. 3a. Clearly, the agreement between the theoretical and measured values for the "bad" specimens is improved in presence of the interfacial layer with a shear wave speed of about one fifth of the Titanium layer.

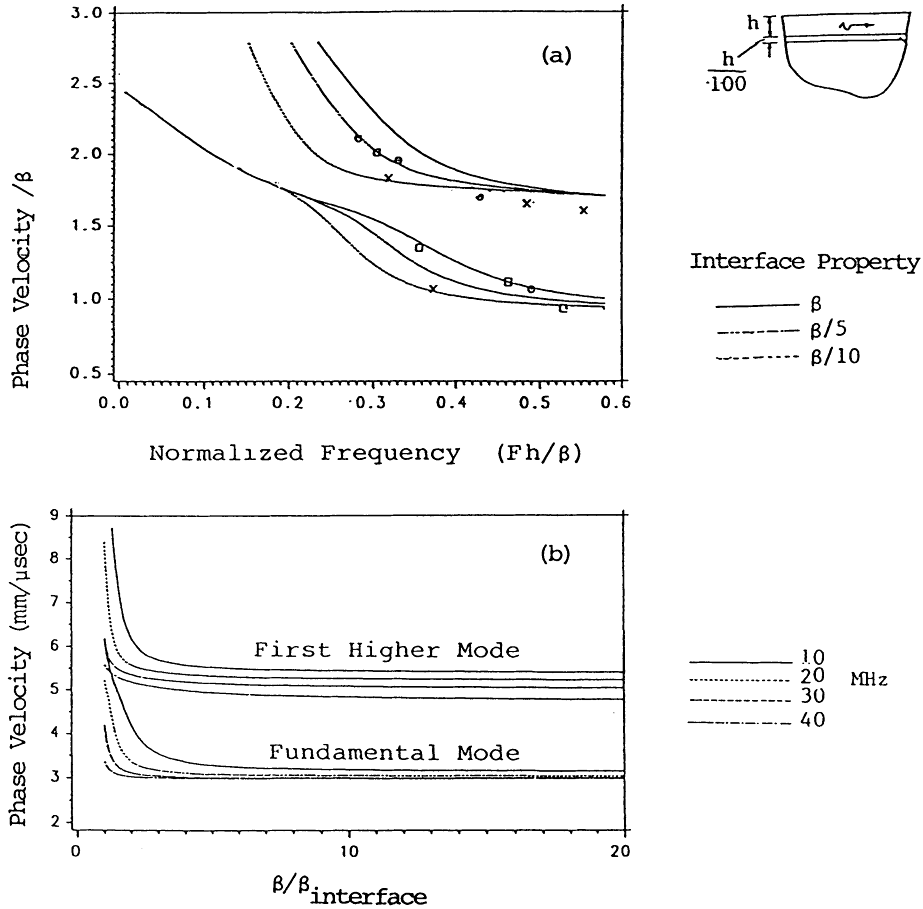

Figure 3. Variation in the dispersion curves with deteriorating bond shear strength

SUMMARY AND CONCLUSIONS

In this paper we have addressed one approach to the determination of bond properties between a metallic layer/substrate combination via the reflection acoustic microscope. Rayleigh waves that are readily generated in the acoustic microscope appear to be well suited for a fundamental understanding of adhesion in layered structures.

Specially prepared layered metallic samples to enhance or reduce adhesion were examined experimentally in a wide-band low-frequency acoustic microscope. The examination involved the measurement of Rayleigh wave dispersion over a wide frequency range. Substantial differences in the dispersion was observed and tentatively related to the bond properties between layer and substrate. The possibility of observing a higher order (Sezawa) mode that might be excited simultaneously with the fundamental (Rayleigh) mode was considered. It is not clear at present as to why the measured velocities in the bad specimens are closer to the second mode rather than the fundamental. The anomalous behavior of one data point for each specimen also remains unexplained. Further research is currently in progress in an effort to understand these features of the data.

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